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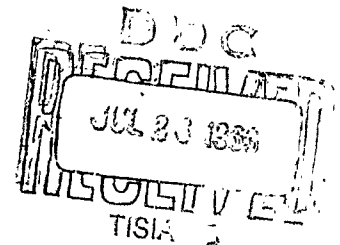CAMERON STATION, ALEXANDRIA, VIRGINIA

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INFLUENCE OF GRAIN BOUNDARIES ON HIGH TEMPERATURE STRENGTH OF POLYCRYSTALLINE SOLIDS

BY

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INFLUENCE OF GRAIN BOUNDARIES ON HIGH TEMPERATURE

STRENGTH OF POLYCRYSTALLINE SOLIDS

by

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DMS Report No. 63-7

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ABSTRACT

Grain boundaries may influence high temperature strength in many ways: as barriers to dislocation motion, as sources or sinks of vacancies and dislocations, and by grain boundary shearing. In all of these considerations the type of boundary must be taken into account. Experimental results on the influence of grain size on creep of aluminum suggest why coarse grain materials are often stronger in their steady state creep resistance over fine grain materials. As-recrystallized fine grain aluminum is weak because it usually contains high angle random boundaries and as such can easily deform by boundary shearing; in addition, such boundaries may be good sources or sinks of dislocations and vacancies. On the other hand, coarse grain aluminum is strong because it develops subgrains during creep; the subgrain boundaries, which consist of dislocations, are good barriers to dislocations, and these boundaries do not exhibit shearing. The strength of the subgrain boundary formed during creep of coarse grain aluminum appears to be strongly dependent on purity; addition of 250 atom parts per million iron increases the steady state creep resistance of coarse grain aluminum by one thousand times at 400° C.

INFLUENCE OF GRAIN BOUNDARIES ON HIGH TEMPERATURE

STRENGTH OF POLYCRYSTALLINE SOLIDS

Introduction

At high temperatures at least five characteristics of grain bound-
aries are important in determining the strength and ductility of a poly-
crystalline aggregate: (1) Grain boundaries may contribute to deforma-
tion by grain boundary shearing. An example of extensive grain boundary
shearing during creep of a coarse grain aluminum at elevated temperature[1]
is illustrated in Figure 1. The grid network, made by machine ruling, is
clearly displaced at the grain boundaries after creep. (2) Grain bound-
aries may be sources of vacancies and thereby assist in the process of dis-
location climb. Barnes[2] and Barnes, Redding and Cottrell[3] have per-
formed classical experiments revealing the possible role of grain boundar-
ies as sources of vacancies. The type of experiment performed is illus-
trated schematically in Fig. 2. These authors irradiated a polycrystalline
copper block with alpha particles (helium atoms). These particles have the
property of penetrating the copper lattice and locating interstitially to
form a helium rich region. This supersaturated band is more resistant to
etching than the surrounding pure copper and is thus readily observed
metallographically. If the irradiated specimen is annealed at 800° C,
bubbles of helium are observed to have formed either near grain boundaries
or near the surface. The results suggest that grain boundaries and sur-
faces are good sources of vacancies; it is believed that these vacancies
migrate to helium atoms and then permit these atoms to diffuse substitution-
ally through the copper lattice to combine and form bubbles. (3) Grain

Fig.I   SHEAR ALONG A GRAIN BOUNDARY IN HIGH
PURITY ALUMINUM.
CREEP STRESS = 250 PSI, ε_t = 0.07, T = 610 °K,
(after FAZAN, SHERBY and DORN, 1954).

COPPER BLOCK BOMBARDED BY ALPHA PARTICLES
(He ATOMS)

↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓

HELIUM-RICH REGION

IRRADIATED COPPER ANNEALED AT 800°C

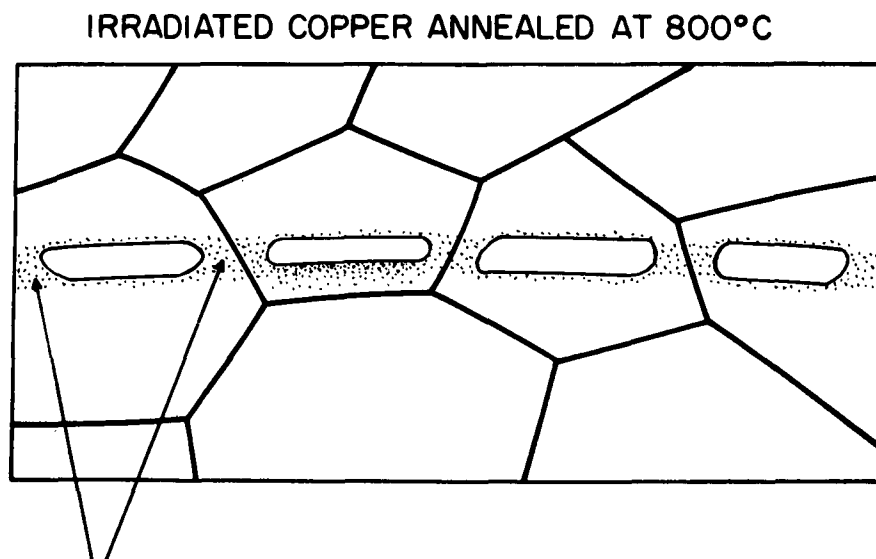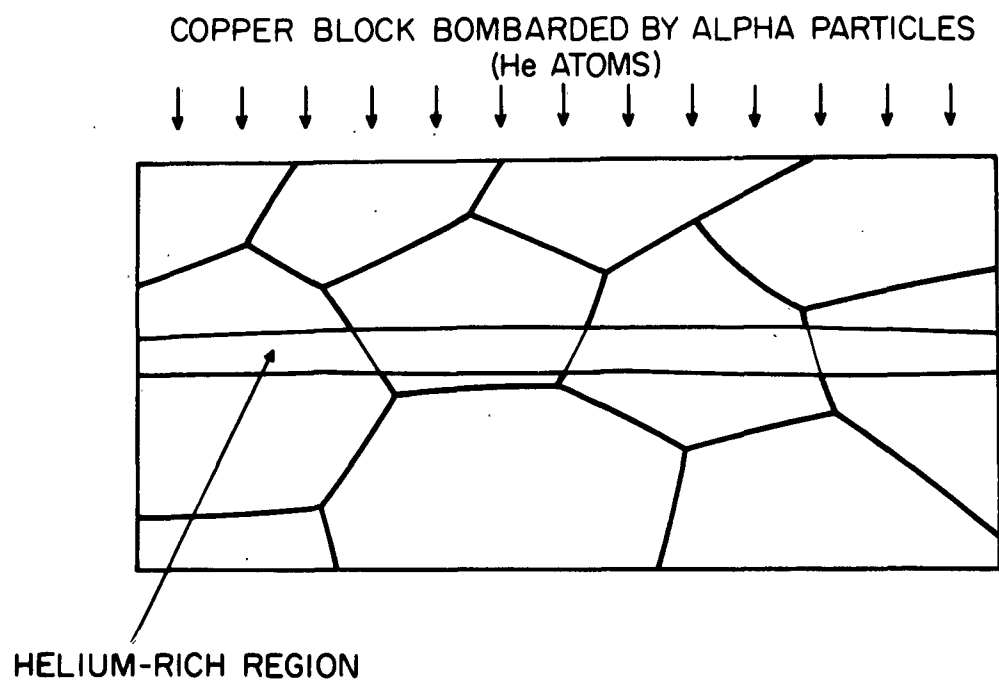Fig.2 HELIUM GAS BUBBLES FIRST FORM PREDOMINATELY
       NEAR GRAIN BOUNDARIES AND SURFACES. THESE OB-
       SERVATIONS SUGGEST THAT GRAIN BOUNDARIES AND
       SURFACES ARE GOOD SOURCES OF VACANCIES; VACAN-
       CIES MIGRATE TO He ATOMS AND PERMIT THESE ATOMS
       TO MIGRATE SUBSTITUTIONALLY THROUGH THE COP-
       PER LATTICE   TO COMBINE AND FORM VOIDS (after
       R.S. BARNES, 1958).

boundaries may be sources of dislocations. Recent studies with electron transmission microscopy have revealed that grain boundaries and surfaces often reveal the property of dislocation emission. An example of dislocations emitted from grain boundaries is shown in Fig. 3 from the work of Szirmae and Fisher[4]. Although this example represents deformation at low temperature it would appear reasonable to assume that such behavior would occur equally well at high temperatures. (4) Grain boundaries may migrate under stress. An example of alternate grain boundary shearing and grain boundary migration is given in Fig. 4 for pure aluminum[1]. Grain boundary migration may relieve the high stresses present at grain boundaries after shearing has taken place and thus permit further grain boundary shearing. This type of deformation is believed, by some, to be a major contributor to deformation at elevated temperature.[5, 6] (5) Grain boundaries may be barriers to dislocation motion. An example of the great deal of activity present near grain boundaries as revealed by etch pitch analysis is shown in Fig. 5 for an Fe-Si alloy deformed by creep at 600° C.[7] The fine lines shown almost parallel to the grain boundaries and at right angles to the $1\bar{1}1$ slip direction are believed to be polygonized walls of dislocations. A higher magnification of a similar region, Fig. 6, clearly reveals the gradual formation of the dislocation walls from separate dislocations as well as the piled-up nature of the walls in the vicinity of the grain boundary.

The first four factors mentioned above probably contribute to the weakening of a material at elevated temperature whereas the last-mentioned factor contributes to strengthening. In each of these contributions to mechanical properties the type of boundary must be taken into account. For
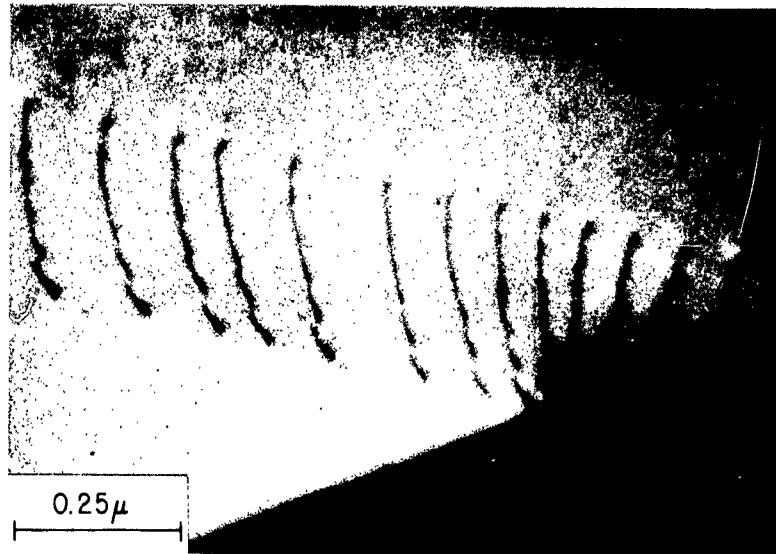
Fig. 3 DISLOCATIONS PROPAGATING FROM A
GRAIN BOUNDARY IN IRON CONTAINING
18 % Cr AND 12 % Ni (after R.M. FISHER
and A. SZIRMAE, 1959).
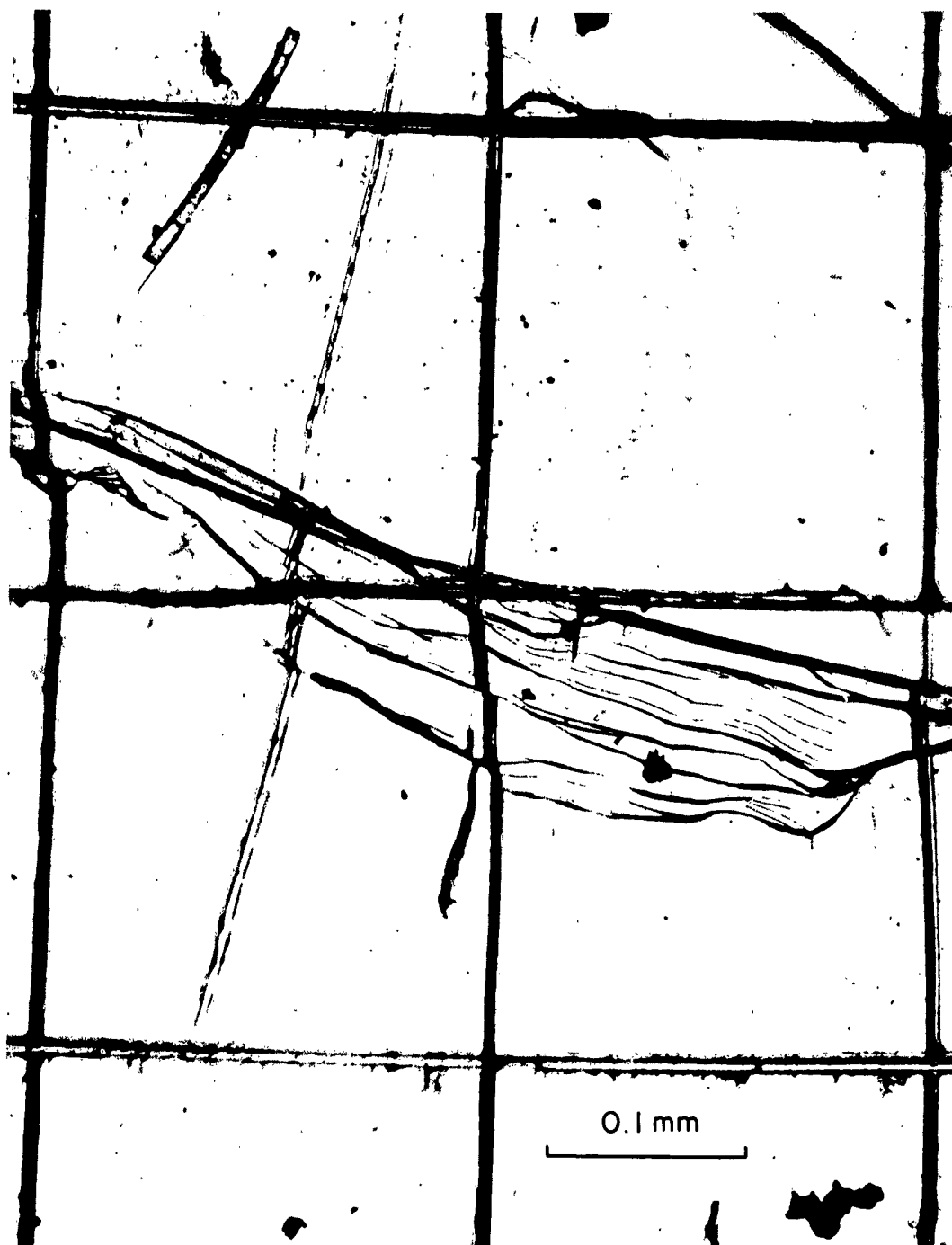
Fig. 4 MULTIPLE BOUNDARY SHEARING AND MIGRATION
IN HIGH PURITY ALUMINUM.
CREEP STRESS = 250 PSI, $\varepsilon_t = 0.07$, T = 747 $^\circ$K,

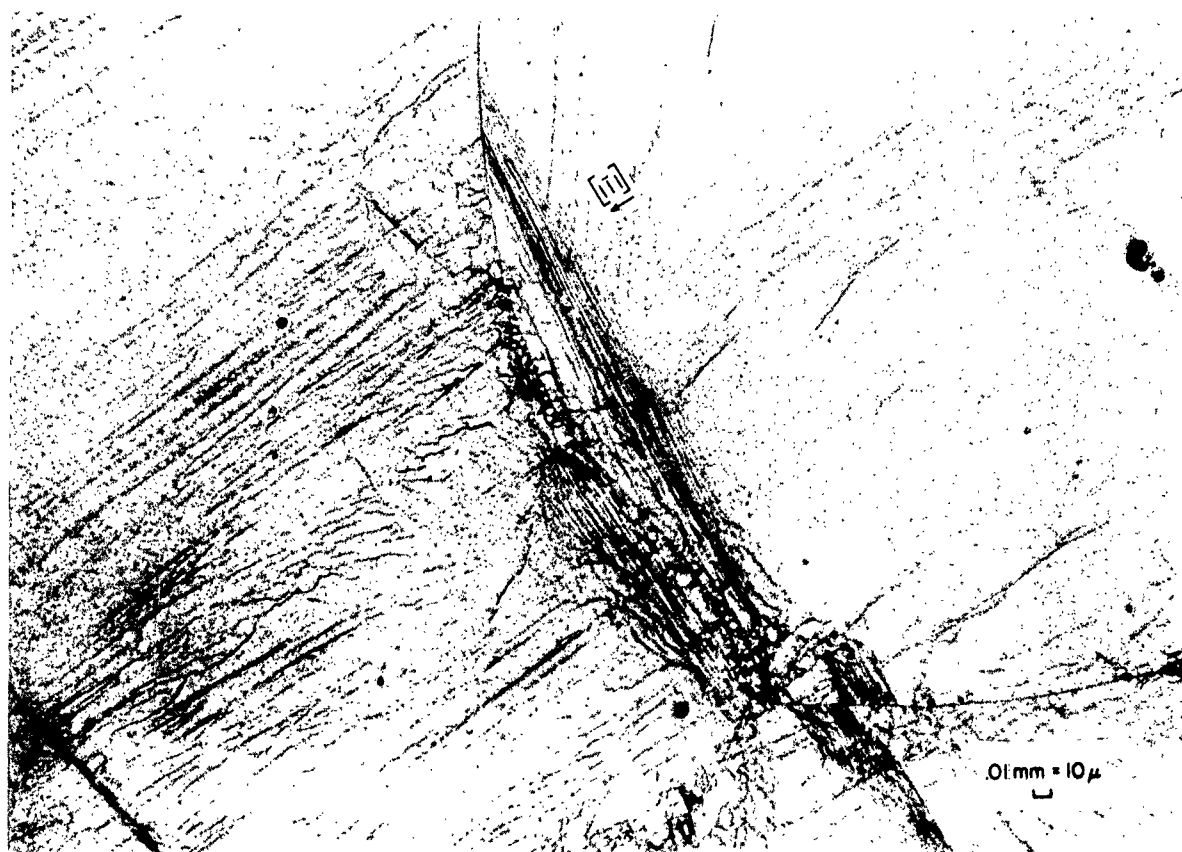(after FAZAN, SHERBY and DORN, 1954).

Fig. 5 STRUCTURE OF Fe- 3 % Si ALLOY AFTER CREEP AT σ = 5450 psi AT 600°C TO TENSILE STRAIN OF 0.044.
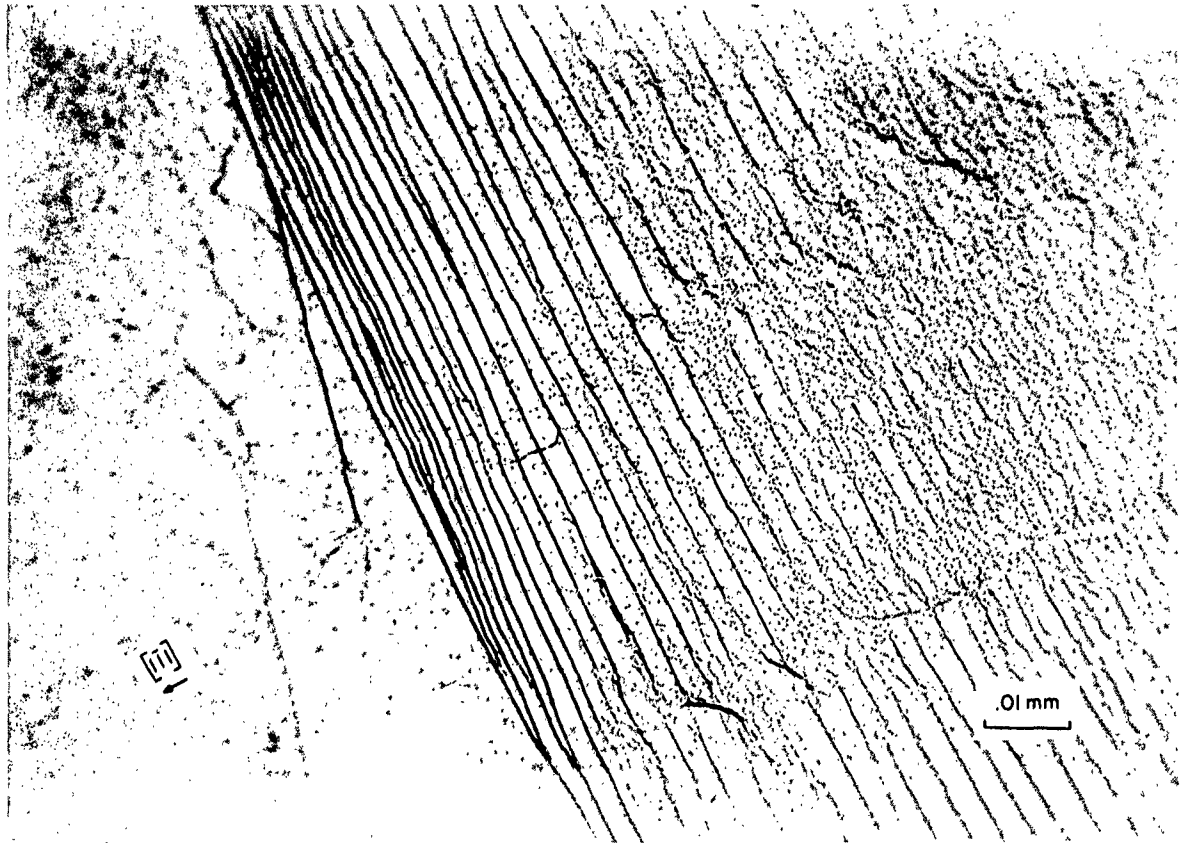STRUCTURE REVEALED BY ETCH PIT TECHNIQUES (after J. L. LYTTON, 1962).

Fig.6 SAME AS PREVIOUS PHOTOMICROGRAPH EXCEPT AT HIGHER MAGNIFICATION (after J.L LYTTON, 1962).

example, a low-angle boundary might be expected to be a poor source of va-
cancies, a poor barrier to dislocation motion, but exhibit no grain bound-
ary shearing. An intermediate angle grain boundary may also be a poor source
of vacancies, may not be able to shear, but may be a better barrier to dis-
locations. A high-angle boundary, on the other hand, may be a good source
for vacancies, may exhibit great ease in grain boundary shearing, but may
be a good barrier to moving dislocations.

Current Status of Grain Boundary Contribution to High Temperature Strength

The strength of polycrystalline pure metals above 0.5 of the absolute
melting temperature has been studied both phenomenologically[8] and theoret-
ically[9-11]. Two major factors influencing the high temperature strength
of solids are the diffusion rate of atoms and the elastic modulus. Over a
wide range of strain rates and temperatures the strength (at a given strain
rate) is inversely proportional to the fifth root of the atomic diffusivity,
D, and directly proportional to the modulus, E. It is believed that the
diffusivity is important because it controls the rate at which dislocation
climb occurs. The elastic properties are important because they influence
the size of the stress fields which form barriers to moving dislocations and
determine the basic nature of dislocations and dislocation interactions.

In analyzing the high temperature creep behavior of pure polycrystal-
line metals[8], it was shown that an excellent correlation was found to
exist between the steady state creep rate and the grain size (for constant
D and constant ratio of stress over elastic modulus, $\frac{\sigma}{E}$). This correlation
is shown in Fig. 7. One is tempted to conclude from these results that
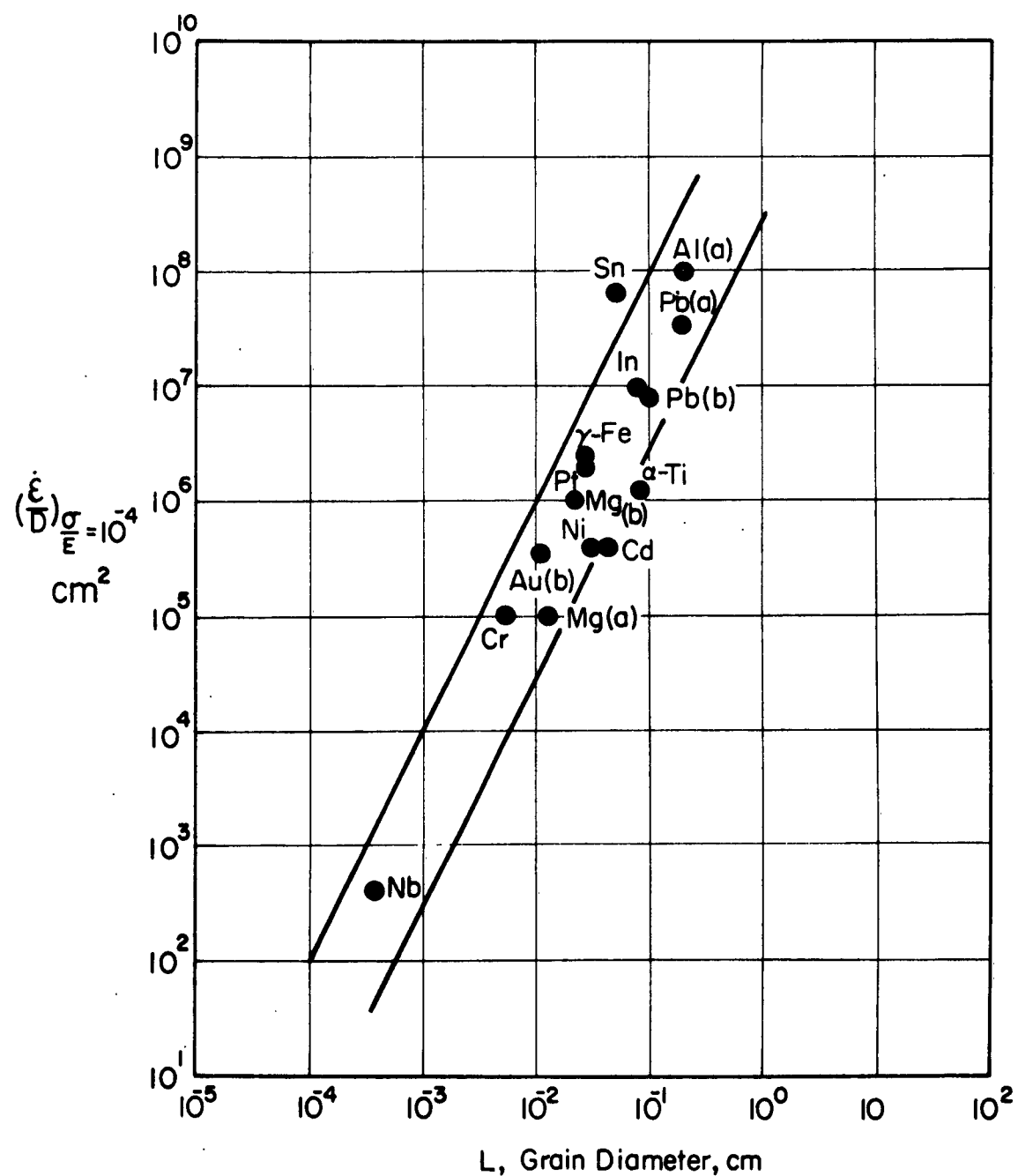
Fig. 7 POSSIBLE INFLUENCE OF GRAIN SIZE ON THE HIGH
TEMPERATURE CREEP RESISTANCE OF PURE METALS
(COMPARED AT THE SAME DIFFUSION RATE AND SAME
VALUE OF CREEP STRESS OVER ELASTIC MODULUS)

grain boundaries are good barriers to dislocation motion at high temperature since decreasing the grain size leads to increasing the creep resistance. Weertman's first theory of creep based on dislocation climb[9] predicted a steady state creep rate proportional to LL' where L was the grain size and L' the subgrain size; this theoretical relation closely approaches the phenomenological observation of $\dot{\mathcal{E}} = KL^2$ obtained from Fig. 7.

The above-cited results imply that the best material for high temperature strength would be one that has a low atomic mobility (therefore high melting temperature, close-packed structure and high valence state[12]), high elastic modulus and fine "stable" grain size. The quantitative relation between creep rate and grain size given in Fig. 7 suggests a strong influence of grain boundaries on high temperature strength; if the grain size can be reduced by a factor of ten the creep rate will diminish by a factor of one hundred. As encouraging as this conclusion may be, there are many anomalies in the literature which are not in agreement with this finding. Three major observations have been made which tend to yield different conclusions on the contribution of grain boundaries to high temperature strength. These observations are discussed in the following sections.

Grain Boundary Shearing. It has been well established that grain boundary shearing does take place during creep at elevated temperatures. If this mechanism is rate controlling it would be anticipated that a coarse grain material (few grain boundaries) would be more creep resistant than a fine grain material. The importance of this deformation mechanism to creep at high temperatures has not been resolved. The majority of investigators[1, 13, 14] have suggested that the contribution by grain boundary shearing

never exceeds 10 to 15%. Rachinger[5, 6], on the other hand, working with aluminum polycrystals, obtained results which seem to suggest that grain boundary shearing may contribute in a major way to high temperature deformation. He felt that most investigators had obtained anomalous results because they had evaluated the amount of grain boundary shearing by surface observations only as in Figs. 1 and 4. Rachinger sectioned his specimens after high temperature deformation and by observing the shape of interior grains in his material concluded that grain boundary shearing contributed to almost all the deformation observed; that is, the grains retained their original shape. A possible explanation as to why surface grains behave so differently from interior grains of a polycrystalline aggregate may be due to the fact that slip can occur readily in the former because the external surface offers little resistance to dislocation motion. Observation of the shape of interior grains after creep in iron was recently done by Garofalo, et al[15, 16]. Their observations seem to suggest, as in Rachinger's work, large contributions to plastic flow by grain boundary shearing. Further effort to clarify the importance of grain boundary shearing, especially within the bulk of the material, is certainly needed.

Optimum Grain Size Effect. A review of the literature on grain size effects on creep reveals, in at least two instances[17, 18], an optimum grain size for maximum creep strength. Increasing or decreasing the grain size beyond this critical size increases the steady state creep rate. An example of this is shown in Fig. 8 for Monel metal and tin.

In the coarse grain size range the results can be explained, as previously interpreted, by assuming that the grain boundaries are good barriers
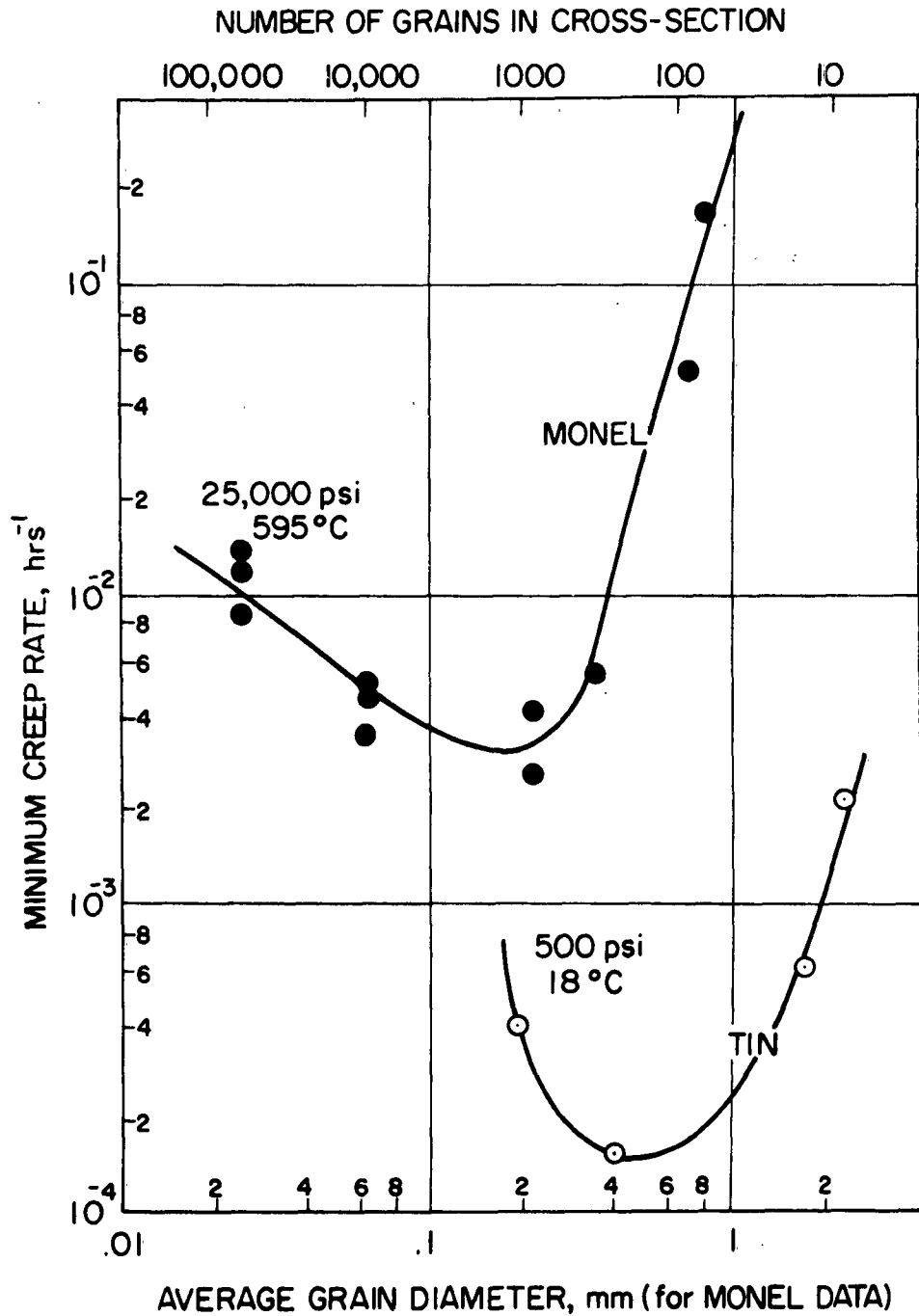
Fig. 8 INFLUENCE OF GRAIN SIZE ON CREEP OF MO-
NEL AND TIN (after SHAHINIAN and LANE,
1953 and HANSON, 1939).

to dislocation motion; hence, the coarser the grain size, the greater the distance between barriers, and the faster the creep rate. On the other hand, the results in the coarse grain size range may be complicated by the fact that the ratio of external surface area to grain boundary area is high; thus, the external surface may play an important part in determining the creep resistance in this range. This explanation, however, would hardly seem likely for Monel metal since the coarsest grain size used yielded over 50 grains in the cross-section (Fig. 8).

In the range of fine grain size the data can be explained either by assuming that grain boundary shearing is important or that grain boundaries are good sources of vacancies or dislocations. On the other hand, these fine grain materials may have been unstable during creep and grain growth may have contributed to the rapid rates of creep observed. Parker has suggested yet another explanation[19, 20]. He feels that the majority of grain size studies on creep have been incorrectly performed. It is usual to prepare various grain sizes by annealing a given cold-worked material at successively higher annealing temperatures. In the as-recrystallized state, there exists in the polycrystalline aggregate boundaries of various degrees of misorientation. During grain growth, however, the high energy (high angle) boundaries tend to be eliminated more readily than the low energy (low angle) ones; hence the coarse grain samples will not only have large grains, but may also have a preponderance of low angle boundaries. Thus, two things are altered with increased annealing temperature: the grain size and the type of grain boundary. Parker reasoned that the coarse grain material may be more creep resistant than the fine grain material not so much because it had

large grains, but because it had primarily low-angle boundaries which are poorer sources of vacancies, thus leading to a low dislocation climb rate. Besides the problem of controlling the type of grain boundary another variable is introduced by the usual method of preparing specimens of different grain sizes. Higher annealing temperatures result in a greater dissolution of the impurity atoms contained in the material. These impurity atoms will precipitate upon subsequent cooling. Thus, a different distribution of precipitates may be present in a coarse grain material (high annealing temperature) than in a fine grain material (low annealing temperature). Additional work is very necessary to clarify these various issues.

Deformation by Mass Migration of Atoms. Nabarro[21] and Herring[22] both derived a creep relation based on the mass diffusion of atoms from the sides of a grain to the top and bottom surfaces of a grain when under a tensile stress. In addition to predicting a linear relation between stress and creep rate, the authors showed that the steady state creep rate was inversely proportional to the grain diameter squared so that smaller grains lead to higher creep rates. Experimental verification for the Nabarro-Herring mechanism is limited for pure metals. It has been clearly established that if this creep mechanism is ever valid it is only so at very low stresses near the melting temperature. Only face-centered cubic Cu, Au and Al have been studied at sufficiently low stresses to test the Nabarro-Herring mechanism. Fig. 9 shows the good correlation obtained with the Cu and Au data and the poor correlation with Al.

It is believed by some[23-25] that the Nabarro-Herring creep mechanism is important in the creep of crystalline oxides whereas others[26]
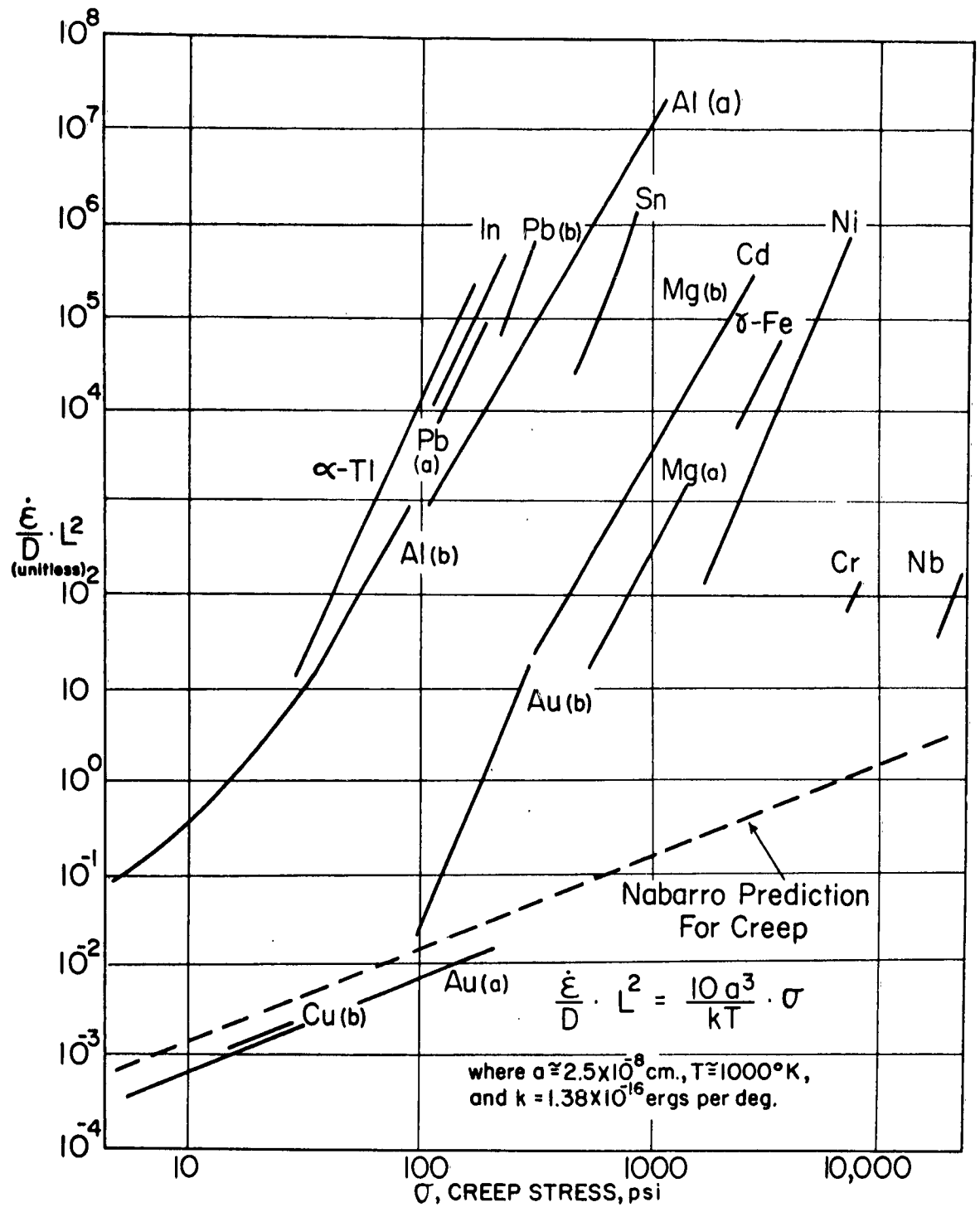
Fig.9 Comparison of creep behavior of various pure polycrystalline metals with Nabarro diffusional creep theory (data obtained from references given in ref. 8).

have suggested that grain boundary shearing predominates as the deformation mechanism in such materials. Folweiler appears to have shown a linear dependence of strain rate to stress for creep of polycrystalline alumina over a wide stress range (500 to 10,000 psi) and a grain size dependence on creep as predicted by the N-H relation. It is to be pointed out, however, that interpretation of the results must be subject to considerable question for the material exhibited a great deal of cracking during creep. It is also very likely that steady state creep rates were not obtained especially at the low stresses. Further, the absolute rate of creep is more nearly equal to that predicted by a dislocation-climb mechanism rather than by the Nabarro-Herring mechanism as can be seen in Fig. 10. This point was ignored by Folweiler. More recent tests performed by Warshaw and Norton[24] seem to partially confirm the results of Folweiler although the new work reveals that when the material contains coarse grains it does not show a linear dependence of creep rate on stress. Other tests performed by Beauchamp, Baker and Gibbs[25] are too limited to help clarify the mechanism of creep in polycrystalline alumina. All three groups of investigators did their tests in bending where the stress was not constant in the specimen and furthermore, the stress distribution changed with creep straining. Constant stress creep tests should be performed on polycrystalline alumina to shed further light on the creep of this material.

It is clearly evident from the above discussion that the influence of grain boundaries on the high temperature deformation of polycrystalline metals and oxides is far from a settled issue.
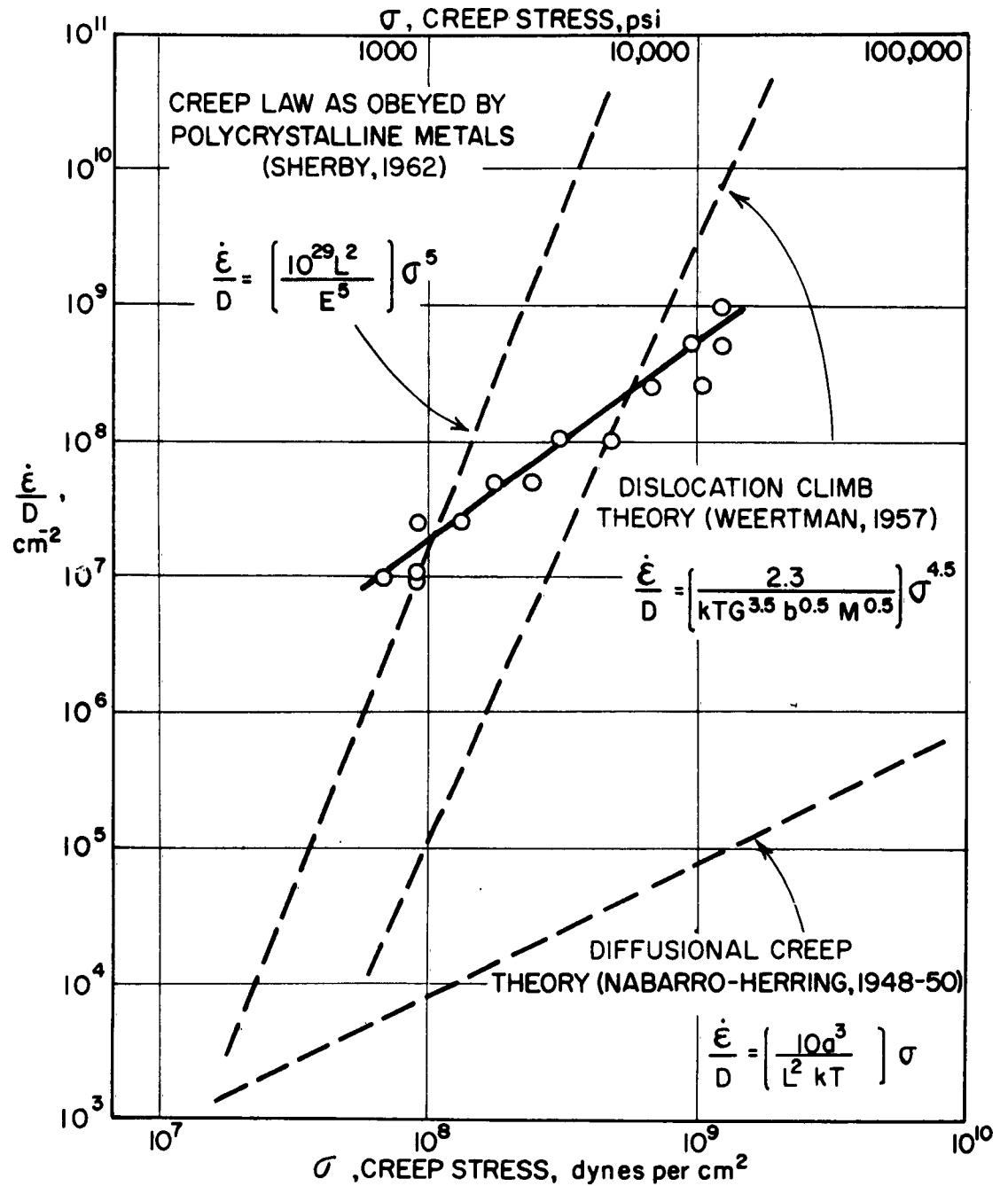
Fig. 10. Comparison of various creep theories with creep of polycrystalline alumina of grain dia-meter, $L = .0034$ cm, at 1800° C (data of Folweiler, ref. 23) $\dot{\epsilon}$ = creep rate, D = volume diffusion coefficient of oxygen in alumina ($D = 10^{11}$ cm^2/sec at 1800° C from ref. 29), E = Young's modulus (24,000 kg/cm^2 at 1800° C from ref. 30), G = shear modulus (= 0.4E), b = Burger's vector, a = lattice spacing ($b = a = 3 \times 10^{-8}$ cm), and M = number of Frank Read sources per cm^3. M is related to N, the density of dislocations per cm^2, by $M^{1/2} = 0.537 \ N^{3/4}$. N assumed equal to 10^8.

Effect of Grain Size on Creep Resistance of Stable Materials

In the ideal case, the influence of grain size on the creep resist-
ance of a very pure metal should be determined; in this way, the complicat-
ing effects associated with impurity atoms can be eliminated. Unfortun-
ately, as shown by Servi and Grant[27], the difficulty here is that grain
growth occurs readily in such pure metals when under the influence of con-
current deformation at elevated temperatures. It was therefore decided to
perform studies on a pure metal doped with a small amount of impurity to
retard grain boundary migration during creep. It is well known that iron
influences aluminum in this manner. High purity aluminum containing 0.054%
Fe (250 atom parts per million iron) was prepared through the courtesy of
the Alcoa Research Laboratories; the only other detectable impurity ele-
ments were magnesium (.004%) and copper (.003%). The material was received
in the form of a cast ingot, two feet long by six inches in diameter. The
as-received cast metal was hot forged into 5/8 inch diameter rods from which
compression creep specimens were machined. Metallographic examination of
the hot forged rods indicated evidence of a heavily worked state in this
condition.

A small grain size was obtained by annealing the creep specimens at
500° C (0.83 Tm) for one hour. The material had an equiaxed grain size
which averaged 0.1 mm in diameter. Longer annealing treatments at 500° C
did not increase the grain size. Furthermore, preliminary creep tests on
this material in the range 250 to 450° C revealed no observable grain growth
during creep. Back reflection Debye-Scherrer photograms revealed no evi-
dence of a preferred orientation texture in the as-recrystallized state.

A large grain size was obtained by annealing the creep specimens at 640° C (0.98 Tm) for approximately 40 hours. Annealing periods in excess of this time did not increase the grain size. The creep specimens annealed in this manner had an equiaxed grain size of 1.2 mm.

Specimens, 0.4" long and 0.3" in diameter were prepared for compression creep testing at 400° C (0.72 Tm). The stress was maintained constant by periodically adding weights during creep testing to correct for the increase in cross-sectional area. The creep specimens were tested in a neutral salt bath (an equal mixture of $NaNO_3$ and $NaNO_2$) for the constant temperature tests; the temperature did not vary by more than $\pm 1°$ C during any given test.

The influence of stress on the steady state creep rate for the two different grain sizes is given in Fig. 11. These data clearly reveal the superior creep resistance of the coarse grain size material. A first consideration would suggest that grain boundary shearing might be an important factor in contributing to the results obtained. The actual creep curves obtained, however, indicate very dissimilar trends for the two grain size materials, Fig. 12. As can be seen in this figure, the fine grain size material exhibited steady state creep characteristics right from the very beginning of the test. On the other hand, the coarse grain size material behaves entirely differently. It exhibits a very rapid rate of creep initially (greater than the fine grain size) but with continued creep straining the creep rate decreases rapidly until finally, at large strains, the creep rate is over 100 times slower than the fine grain material. Obviously, the structure responsible for creep resistance is changing signifi-
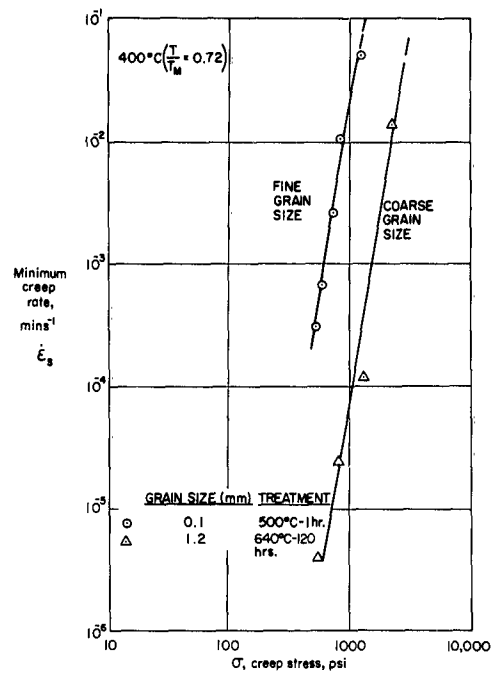
Fig.11 INFLUENCE OF GRAIN SIZE ON THE CREEP RE-
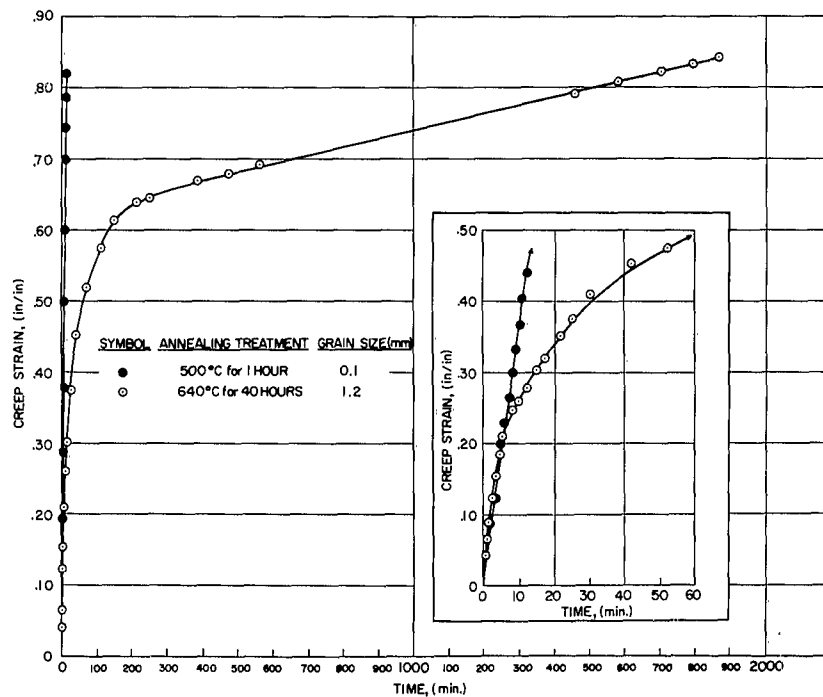SISTANCE OF PURE ALUMINUM CONTAINING
0.05% IRON.

Fig.12 INFLUENCE OF GRAIN SIZE ON THE CREEP CURVE OF PURE ALUMINUM CONTAINING 0.05% IRON AT 400°C
AND 1250 psi.

cantly during creep of the coarse grain material since the creep rate is changing so drastically. In order to determine the changes in structure occurring during creep, deformed specimens were sectioned, electrolytic polished and then anodic film etched. Such preparation permitted a study of the structure of interior grains; furthermore, with the use of polarized light it was possible to clearly identify the subgrain structure that was developed by creep.

Fig. 13 reveals the change in structure that took place during creep of the coarse grain material. As can be seen the formation of subgrains occurred early during primary creep. The surprising result was that the <u>subgrain dimensions were of a given size early during primary creep and remained this same size with further creep straining</u>. The relation of the creep curve to the corresponding structure is interpreted as follows: The subgrains first formed are very low angle sub-boundaries and as such are not good barriers to dislocations. With increased straining the angle of the boundary increases and consequently such boundaries become better barriers to subsequently generated and moving dislocations. Slip line tracing studies after room temperature deformation of the crept specimens revealed that the misorientation of the subgrains did indeed increase with creep straining. After creep of about 0.70 - 0.80 strain, the misorientation was typically between 10 and 15°.

The subgrain size developed during creep (at 1250 psi) of the coarse grain size material is 0.05 mm. This size is not very different from the size of the fine grain material. Why then is there such a large difference in creep resistance between the two materials? The answer may

0.1 mm

BEFORE CREEP

$\varepsilon = 0.28$

$\varepsilon = 0.45$

$\varepsilon = 0.57$

$\varepsilon = 0.85$

Fig.13   DEVELOPMENT OF SUBGRAIN STRUCTURE DURING CREEP OF COARSE GRAIN
         ALUMINUM CONTAINING .05% IRON AT 400°C (σ =1250 psi) (after BUSBOOM,
         1963).

lie in the type of grain boundary present in the fine grain size material.
It is believed they are predominantly high angle-non-dislocation boundar-
ies. As such, grain boundary shearing may be an important mechanism of
deformation, or perhaps such high angle boundaries may be good sinks for
dislocations or good sources of vacancies. Photomicrographs of the fine
grain material after creep at 400° C and σ = 1250 psi are shown in Fig.
14. As can be seen, some subgrain formation is evident but the major
observation is that the grain shapes are not nearly as flattened as one
would expect on the basis that grain boundary shearing was negligible.
These results are interpreted to indicate a large contribution of grain
boundary shearing to creep. In fact, when the Rachinger[5] grain-shape
analysis was used, the contribution to grain boundary shearing was cal-
culated to be over 50% at 1250 psi; at lower creep stresses the contribu-
tion by boundary shearing was even higher.

Servi and Grant revealed a unique correlation between subgrain size
(or slip band spacing) and the creep stress for coarse grain pure aluminum.
Their results are plotted in Fig. 15. As can be seen, the slip band spac-
ing, or subgrain size, developed during creep is diminished with increas-
ing creep stress. These results were shown to be independent of the creep
temperature. In addition, the influence of impurity content is seen to be
negligible since the Al alloy containing .05% Fe used in this investiga-
tion superimposes on the data obtained by Servi and Grant. In spite of
this interesting correlation, however, there is a very significant differ-
ence in behavior between coarse grain high purity aluminum and coarse
grain impure aluminum (Al containing .05% Fe). This difference in creep

0.1 mm
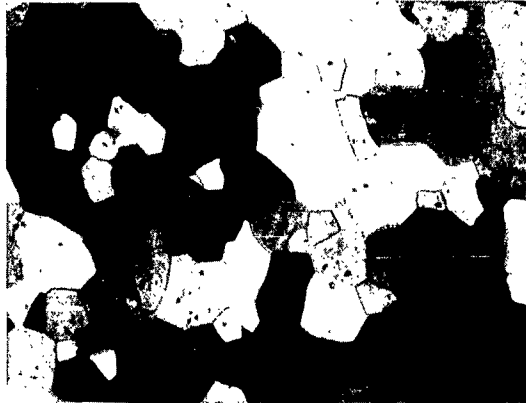
BEFORE CREEP
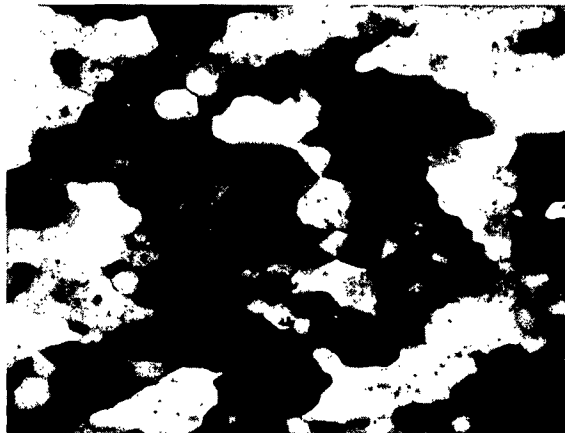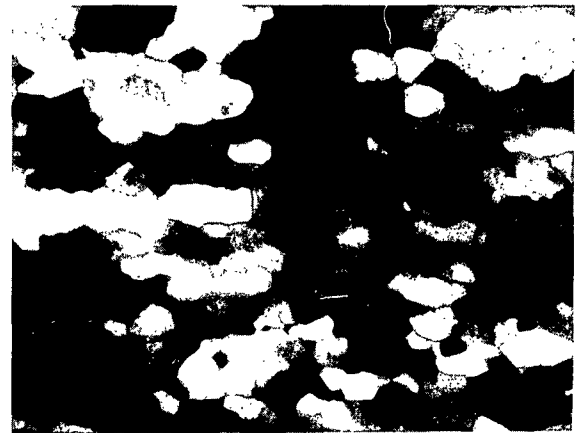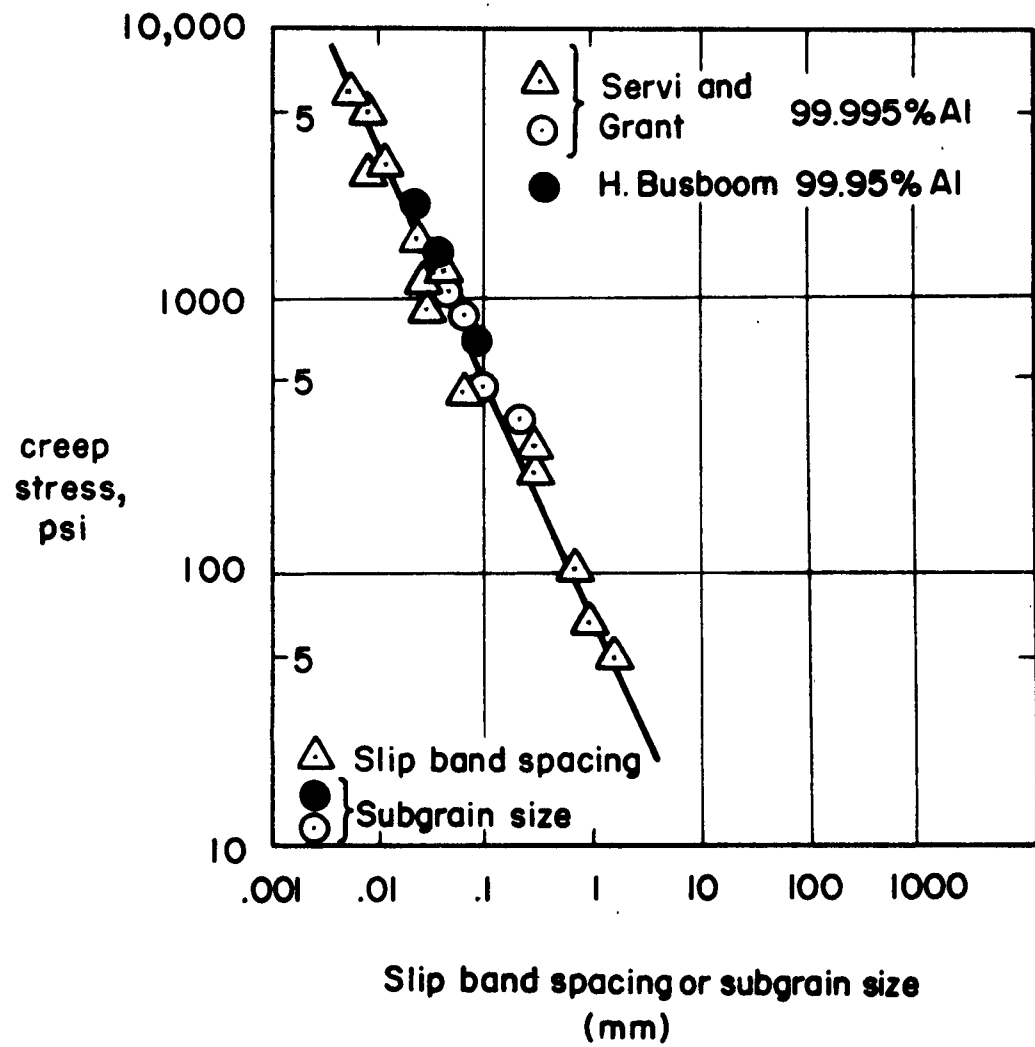
$\varepsilon = 0.46$ $\varepsilon = 0.85$

Fig.14   STRUCTURE OF FINE GRAIN ALUMINUM CONTAINING .05% IRON AFTER
CREEP AT 400°C (σ=1250 psi) (after BUSBOOM, 1963).

Fig.15 INFLUENCE OF CREEP STRESS ON SUB-
GRAIN SIZE FOR COARSE GRAIN ALUM-
INUM.

resistance is shown in Fig. 16; the impure aluminum is 1000 times more

creep resistant than the pure aluminum at 450° C. In order to uncover

the cause for this difference in creep behavior, a high purity aluminum

specimen (99.995% Al) was deformed to various strains at 1250 psi.

Photomicrographs of specimens, after deformation at this stress,

are shown in Fig. 17. As can be seen, similar trends are noted in the

pure aluminum as were found for the impure aluminum. At $\varepsilon = 0.24$ the

subgrains are clearly evident although they are not very sharp, and at

$\varepsilon = 0.85$ they are very well defined. The subgrain size in the pure

aluminum is about 0.05 mm which is the same as that observed in the case

of impure aluminum (Fig. 13). Thus, the difference in the steady state

creep resistance between the two aluminum materials cannot be attributed

to the difference in subgrain size developed. Rather, it appears likely

that the high strength of the impure aluminum is due to the presence of

solute Fe atoms or $FeAl_3$ precipitates at the subboundary walls in this

material. These foreign atoms or impurity particles probably prevent

the dislocation boundaries from bowing under stress to allow moving dis-

locations to pass. Impurity particles have not been detected to date at

light microscopic magnifications, but may be too small for observation

in this manner.

SUMMARY

Grain boundaries play an important part in determining the high

temperature strength of polycrystalline solids. Boundaries may influence

the strength in many ways: as barriers to dislocation motion, as sources
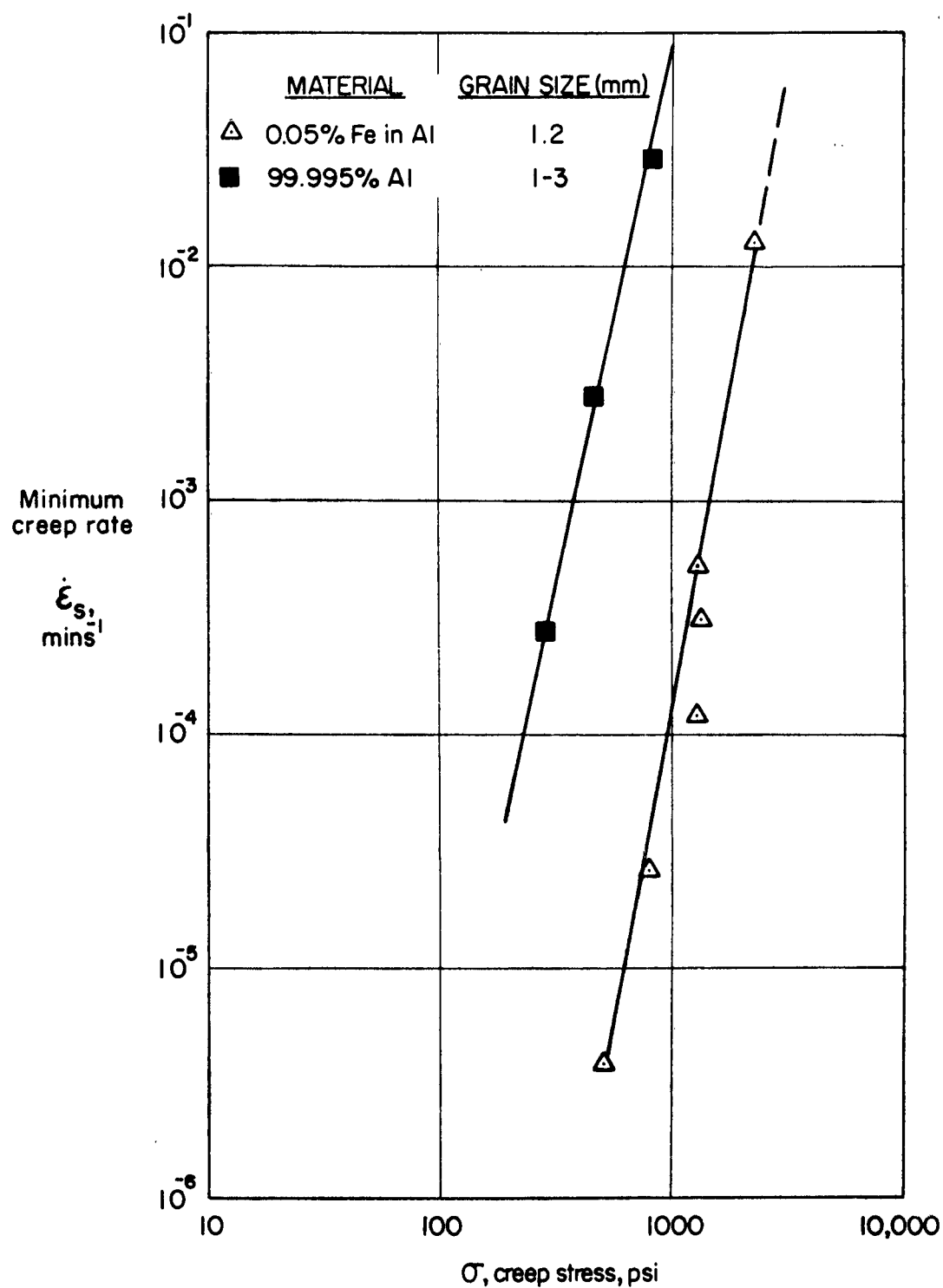
Fig. 16   INFLUENCE OF PURITY ON THE CREEP RESIST-
ANCE OF COARSE GRAIN ALUMINUM AT 400°C.

$\varepsilon = 0.24$

0.1 mm

$\varepsilon = 0.85$

Fig.17 DEVELOPMENT OF SUBGRAIN STRUCTURE DURING CREEP
OF COARSE GRAIN PURE ALUMINUM (99.995%) AT $\sigma = 1250$.
psi.

or sinks of vacancies and dislocations, and by grain boundary shearing. The exact contribution to strength from these various mechanisms has not been clearly resolved to date. In all these considerations the type of boundary present must be taken into account. Analyses of deformation data at high temperature are further complicated by the formation of subgrains; the boundaries of these subgrains consist of a network of dislocations and contribute to increasing the strength of a polycrystalline aggregate. As the angle of the dislocation boundary increases, it becomes a better barrier and the material becomes stronger.

Influence of grain size was studied on the high temperature strength of aluminum containing a small amount of iron (250 atom P.P.M.). The results obtained suggest why coarse grain materials quite often exhibit much greater steady state creep resistance than fine grain materials at elevated temperatures. An as-recrystallized fine grain size material may be weak because it usually contains high angle random boundaries and as such can easily deform by grain boundary shearing (in addition, such boundaries may be good sources or sinks of dislocations and vacancies). On the other hand, a coarse grain size material may be strong because it may develop subgrains during creep; the subgrain boundaries, which consist of dislocations, can be good barriers to dislocations, and these boundaries do not exhibit shearing. This proposal is also consistent with the fact that the initial creep rate of a coarse grain material is greater than a fine grain material, at a given stress and temperature.

The strength of the subgrain boundary formed during creep of coarse

grain aluminum is strongly dependent on purity. Coarse grain impure aluminum containing 250 atom parts per million iron is one thousand times more creep resistance in its steady state creep behavior than high purity aluminum at 400° C. This is believed to be due to the strengthening of the subboundaries by solute atoms or $FeAl_3$ particles. Such dislocation boundaries are probably good barriers to dislocations since the boundaries would be more rigid under stress and would be less easily penetrated by moving dislocations. It is believed that the preparation of such poly-sub-crystalline aggregates can lead to the development of a new series of technologically important creep resistant alloys.

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